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HIGH TEMPERATURE SPECTRAL EMISSIVITY
OF YTTRIUM, SAMARIUM, GADOLINIUM,
ERBIUM AND LUTETIUM OXIDES

By
W. R. McMahon
D. R. Wilder

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January 1963

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Ames Laboratory
at
Iowa State University of Science and Technology
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HIGH TEMPERATURE SPECTRAL EMISSIVITY OF YTTRIUM, SAMARIUM, GADOLINIUM, ERBIUM AND LUTETIUM OXIDES*

W. R. McMahon and D. R. Wilder

ABSTRACT

Spectral emissivities are given for Y_2O_3 , Sm_2O_3 , Gd_2O_3 , Er_2O_3 and Lu_2O_3 in the visible portion of the spectrum for temperatures up to $1600^\circ C$. The data were obtained using a carbon arc image furnace as an energy source, a photovoltaic cell as a radiation detector, a thermocouple as a temperature detector, and a magnesium oxide smoked plate as a standard of reflectance. Emissivity is calculated from a curve of combined reflectance and emitted radiation versus temperature by using Kirchhoff's law and a method of successive approximations to solve Wien's equation.

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INTRODUCTION

A knowledge of the spectral emissivity of materials becomes of increasing importance as the demand for increased efficiencies force utilization temperatures higher and higher. There exists a growing need for accurate data on the emissivity of materials because of the extreme temperatures now commonly encountered in nearly every phase of industrial endeavor.

* This report is based on an M. S. thesis by William Raymond McMahon submitted November, 1962, to Iowa State University, Ames, Iowa. This work was done under contract with the Atomic Energy Commission.

At elevated temperatures thermal emission accounts for a large part of all heat transfer, making knowledge of emissivity essential for pyrometry and heat balance calculations. Under certain conditions, such as those found in outer space, radiant heat transfer is the only method of heating or cooling. In such instances spectral emissivity data is of extreme importance to the designer. Aerodynamic heating on the structures of hypersonic aircraft is another example wherein the importance of emissivity data is clearly evident.

The advent of higher temperatures has precipitated a need for and the introduction of new super refractories. Of the many materials proposed to meet this need, high purity ceramic oxides are most promising. Their high melting point, chemical stability, high strength, ease of fabrication, oxidation resistance, mechanical and electrical properties, and relative low cost strongly suggest that they will be found in many new and critical applications in this era of high temperature technology. The spectral emissivities of these materials must be accurately determined before their ultimate potential can be realized.

FUNDAMENTALS

Kirchoff's Law

Kirchoff's law quantitatively relates the emitting, reflecting and absorbing properties of a body. The law states that a blackbody placed in a constant temperature cavity with opaque walls and maintained in thermal equilibrium with the walls is absorbing radiant energy from its surroundings at exactly the same rate as it is emitting radiant energy. From this relationship was derived the fact that the total emittance of

a nonblackbody at any given temperature is equal to its total absorptance of radiation from a blackbody at the same temperature. Kirchoff's law applies equally well to spectral radiation and it may be shown that for any specimen the spectral emittance equals the spectral absorptance. In equation form Kirchoff's law may be stated as follows:

$$\epsilon + r + t = 1$$

$$\epsilon' + r' = 1$$

$$\epsilon = a$$

$$\epsilon' = a'$$

where

$$\epsilon = \text{emittance}$$

$$\epsilon' = \text{emissivity}$$

$$r = \text{reflectance}$$

$$r' = \text{reflectivity}$$

$$t = \text{transmittance}$$

$$a = \text{absorptance}$$

$$a' = \text{absorptivity}$$

Wien's Equation

Wien's equation is derived from Wien's expression for the spectral distribution of radiation emitting from a blackbody. It is an expression relating the emissivity of a material to its true and apparant temperature. This derivation is shown below.

$$W_{\lambda} = \frac{c_1}{\lambda^5 e^{c_2/\lambda T}}$$

$$W_{\lambda}' = \frac{\epsilon_{\lambda}' c_1}{\lambda^5 e^{c_2/\lambda T}} = \frac{c_1}{\lambda^5 e^{c_2/\lambda T_b}}$$

$$\frac{\epsilon_{\lambda}'}{e^{c_2/\lambda T}} = \frac{1}{e^{c_2/\lambda T_b}}$$

$$\ln \epsilon_{\lambda}' = c_2/\lambda \left(\frac{1}{T} - \frac{1}{T_b} \right)$$

W_{λ} = energy emitted at indicated wavelength by a blackbody

W_{λ}' = energy emitted at indicated wavelength by a nonblackbody

λ = wavelength

T = true temperature

T_b = apparent temperature

c_1 = first radiation constant

c_2 = second radiation constant

ϵ_{λ}' = emissivity at indicated wavelength.

The equations of Kirchoff and Wien are valid for the entire spectrum or for any interval within the spectrum. They are also valid for directional radiation provided the geometry of illumination and viewing is proper.

There are certain aspects of the spectral distribution that must not be overlooked in using the equations of Kirchoff and Wien. By definition emissivity is a ratio of radiant flux from a body to the flux from a blackbody at the same temperature and of the same size. It follows then, that if the equations are to be valid, the radiation impinging upon the specimen must have the spectral characteristics of a blackbody at the same temperature.

The conditions necessary to validate the equations do not preclude their use in computation of emissivity from reflectivity data obtained

when the incident radiation is at a higher level than could be produced by a blackbody of the same size and temperature as the specimen. The use of the equations is permissible because the reflectivity of a specimen is a function of temperature alone and is independent of the method of heating. The equations are applicable to either total or spectral emittance when the corresponding values of reflectance are used. Thus a plot of reflectivity readily yields a plot of emissivity since $t = 0$ and $\epsilon' = 1 - r'$.

C. 11 LITERATURE REVIEW → 12 Emissivity and Emittance

The word "emit" means to send out, therefore, the emissivity or emittance of a material is a measure of its ability to send out radiant energy. Emissivity and emittance are both unitless numbers because their values are determined by the ratio of two entities with the same units. Obviously, since the rate of emission from a nonblackbody can never exceed the rate of emission from a blackbody, this ratio can never exceed the value of one.

For the specified purpose of clarifying work in the area of thermal radiation, Worthing¹ has established terminology and symbolic representations wherein each term or symbol is meaningful and unique. His terminology is widely used in modern literature and will be used here.

Methods of Measuring Spectral Emittance

Direct Techniques. A considerable amount of literature on various methods is available.³⁻⁹ Worthing¹⁰ has used the hole-in-tube method to measure the spectral emittance of materials which are suited to resistance heating. The spectral detector is sighted first upon the blackbody

hole to determine the blackbody temperature of the specimen and then upon the outside wall of the tube to determine the apparent or brightness temperature of the specimen. The spectral emittance of the specimen is related to these two values by Wien's equation:

$$\ln \epsilon_{\lambda} = c_2 / \lambda \left(\frac{1}{T} - \frac{1}{T_b} \right).$$

The methods of Olson and Katz⁵ and McMahon⁶ can also be used to make spectral determinations of emittance by choosing spectral detectors rather than total detectors.

McDonough¹¹ and Richmond¹² have described methods using blackbodies at the same temperatures as the specimens to determine the spectral emittance of materials. Images of a specimen and a blackbody are projected alternately upon the entrance slit of a monochrometer. The intensity of the wavelengths passing through the monochrometer is measured with a suitable detector and the ratio of the two readings becomes the emittance of the sample.

Emissivity Data from Reflectance Measurements. Null and Lozier¹³ have described a method to measure the emissivity of graphite at arc temperatures. Using a double elliptical mirror carbon arc image furnace as a heat source, a burning carbon arc and a standard reflector are placed alternately in the specimen position and are irradiated by the carbon arc image furnace. A system of rotating shutters is used to separate the radiation emitted by the specimen from that reflected by it. With the standard reflector in the specimen position a measurement of the radiation from the furnace is attainable by viewing it with a suitable detector. With the burning carbon arc in the specimen position a measurement of the radiation from the specimen arc and a measurement

of the combined radiation from the specimen arc and radiation reflected by it are attainable. With these three measurements it is a simple matter to determine the reflectivity of the carbon arc and then to use Kirchoff's laws to determine the emissivity.

Ried and McAlister¹⁴ employed reflectance to measure normal spectral emittance using a furnace constructed so a double-beam spectrometer could "look" through an entrance port normally at two different sections of the inner wall of a blackbody cavity. The specimen was maintained at a constant temperature and was mounted flush with the inner wall of one of the viewing areas. The ratio of the readings from the sample and from the wall of the cavity yields the reflectance of the sample.

Various other methods of measuring emissivity and emittance have been described by Worthing and Halliday,² Worthing,¹⁰ and Forsythe.¹⁵

Radiation Detection with Photoelectric Cells

Larsen and Shenk^{16, 17} have described and patented a device to measure temperatures which utilizes a blocking-layer photocell. The device consists of a photocell mounted in a water cooled holder with the cone of radiation impinging upon the face of the photocell being limited by a number of diaphragms in the holder. They found that with a low external resistance (200-300 ohms) the temperature response of the photocell could be expressed as:

$$E = kT^{12.34}$$

where

E = potential drop across the resistance

k = a constant which is variant with cells

T = temperature of blackbody viewed by photocell °K.

Probably the earliest suggestion for the application of photocells for temperature measurement was made by Ives.¹⁸ In 1923 he patented a photoelectric pyrometer.

Harrison¹⁹ points out the potential of photosensitive materials as detectors in radiation pyrometry and gives an excellent mathematical treatment of the problem of variant spectral response in photosensitive materials.

Recent developments in photoelectric detection have been described by Smith.²⁰

Imaging Furnaces

Null and Lozier²¹ have described carbon arc image furnaces and the many possible geometric configurations. Broida²² describes a method of producing intense beams of thermal radiation from a high current carbon arc and a relay-condenser optical system. He describes the spectral distribution of the arc and various methods used in attenuating the beam. Krolak and Davis²³ describe a device to measure the intensity and spectral characteristics of the beam from a carbon arc image furnace. The device is a spectrometer which receives the beam of the imaging furnace after it is reflected from a magnesium oxide smoked plate. The reflection is used to reduce the intensity of the radiation by diffusing the beam.

EQUIPMENT

The heat source employed in this project was a carbon arc image furnace which incorporated a Simplex Series 1900 projection lamp. The reflecting mirror is ellipsoidal and thus has two focal points. The carbon

arc is located at one of the focal points and all of the heat and light originating at the arc and striking the mirror is concentrated at the second focal point. A second ellipsoidal mirror placed in front of the first mirror such that the major axes of the two ellipsoidal sections were coincidental and at a distance such that the two mirrors had a common focal point midway between them. The remaining focal point of the second mirror was the specimen position.

A Weston Photronic cell Model 856YG was used as a radiation detector and was connected in series with an external resistance of sufficient magnitude to give a linear response of voltage drop across the resistor with change in the intensity of impingent radiation.

Temperature measurements were made with a platinum, platinum-rhodium thermocouple.

Spectral measurements are facilitated by using a set of spectral filters. Wratten filters Number 70, 72B, 73, 74, 75 and 76 were used in conjunction with a one-centimeter cell of a 4% copper sulfate solution in water. The latter was used to absorb the infra-red wavelengths which are passed by all of the above filters.

A Hewlett Packard Model 130BR oscilloscope was used for all measurements of electrical output from both the photocell and the thermocouple.

A General Electric tungsten ribbon filament pyrometer lamp was used in calibration of brightness temperature response of the photocell. A General Electric projection lamp was used to determine the linearity of photocell response with changes in the intensity of impingent radiation.

The pyrometer lamp was calibrated with a Leeds and Northrup optical pyrometer which was in turn calibrated against a primary standard.*

A freshly-prepared water-cooled magnesium oxide smoked plate was used as a reflectance standard.²⁴ The reflectance throughout the entire visible spectrum of such a plate is very close to 0.98.²⁵

A schematic diagram of the apparatus is given in Fig. 1. Figure 2 is a photograph showing the optics of the furnace and the concentration of radiant energy at the specimen position. Figure 3 is a drawing of the photocell-solution filter assembly.

CALIBRATION OF EQUIPMENT

To determine the linearity of response of the photocell to the intensity of impinging radiation a device was constructed using a projection lamp mounted on a slide wire resistor as an approximation of a point source of radiation. The photocell was placed at one end of the slide wire. The projection lamp was supplied with a constant six volts and could be moved toward or away from the photocell while in contact with the slide wire resistor. A relatively low alternating potential was applied across the ends of the slide wire. The output of the photocell and the potential drop along the slide wire from the end located at the photocell to the position of the lamp was fed into the oscilloscope. Under this arrangement the trace of the oscilloscope is a plot of output of the photocell versus distance of the source from the photocell.

* Ribbon filament lamp calibrated under test Number G-13681 by the National Bureau of Standards for the Physics Department Instrument Shop of Iowa State University.

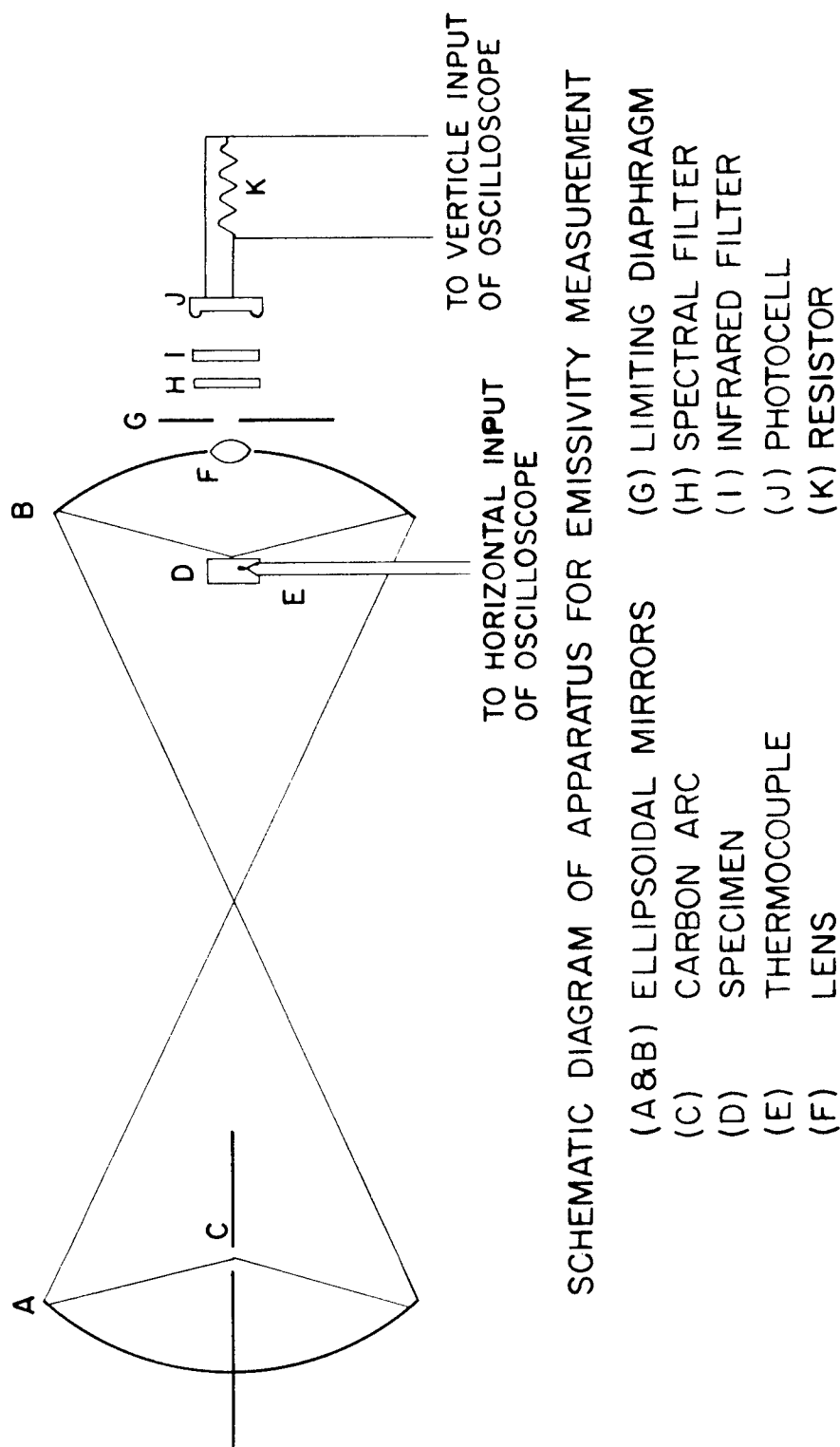


Figure 1. Carbon arc image furnace and measuring circuits.

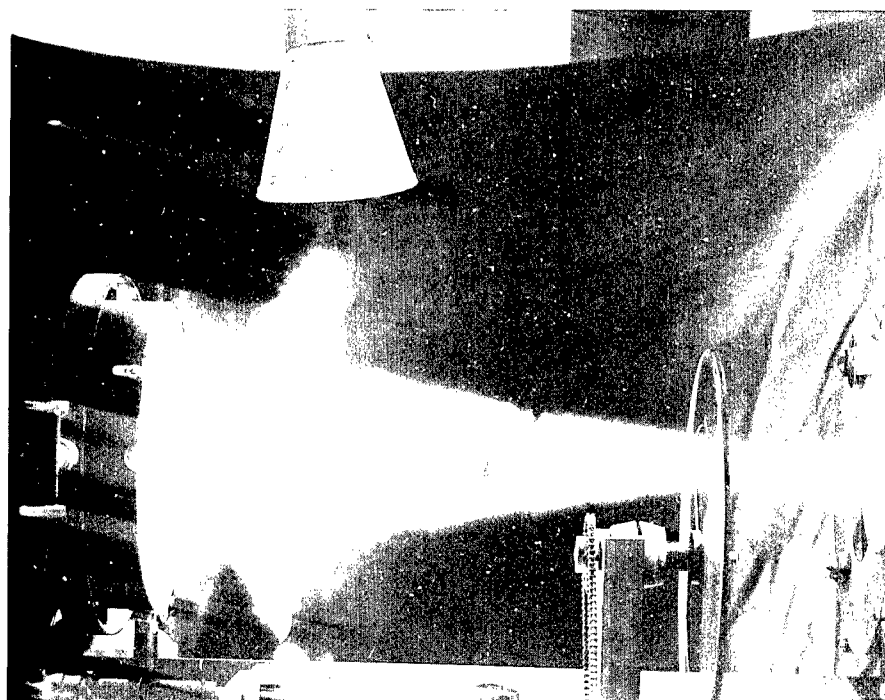


Fig. 2. Photograph showing optical path of carbon arc image furnace.

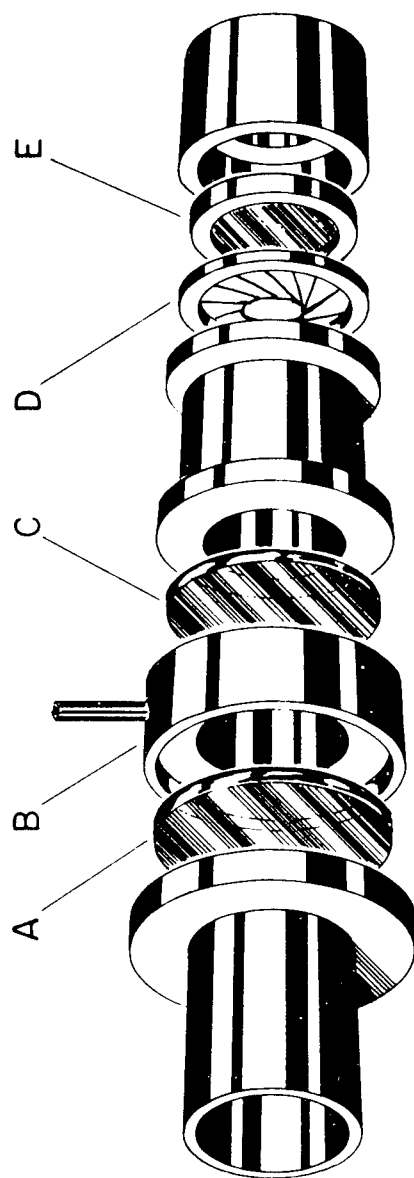


DIAGRAM OF PHOTOCELL HOLDER

- (A & C) GLASS PLATES
- (B) INFRARED ABSORPTION CELL
- (D) LIMITING DIAPHRAGM
- (E) PHOTOCELL

Figure 3. Thermal radiation detector.

The energy from a point source passing through a differential area of a sphere with the source at its center varies inversely with the square of the radius of the sphere. Thus, for a linear response of the photocell, the trace of the oscilloscope must be a plot which is represented by an equation of the form:

$$V = a/X^2$$

where

V = photocell output

a = a constant

X = distance from lamp to photocell.

Variable resistors were introduced into the external circuit of the photocell and adjusted until the response proved to be linear. A different resistance was required for each wavelength and varied in magnitude from 43K ohms to 2 megohms (the input impedance of the oscilloscope). A schematic diagram of the apparatus and the external circuit is given in Fig. 4. A typical calibration curve showing the linearity of response of the photocell to the intensity of radiation passing each of the filters is given in Fig. 5.

The calibrated tungsten filament pyrometer lamp was used to ascertain the brightness temperature response of the photocell. The alternating voltage supplied to the lamp was also fed into the oscilloscope thereby indicating the brightness temperature of the lamp. The lamp filament was placed in the specimen position and its image focused on the photocell. Also, the output of the photocell was fed into the oscilloscope thus producing a plot of output of the photocell versus brightness temperature

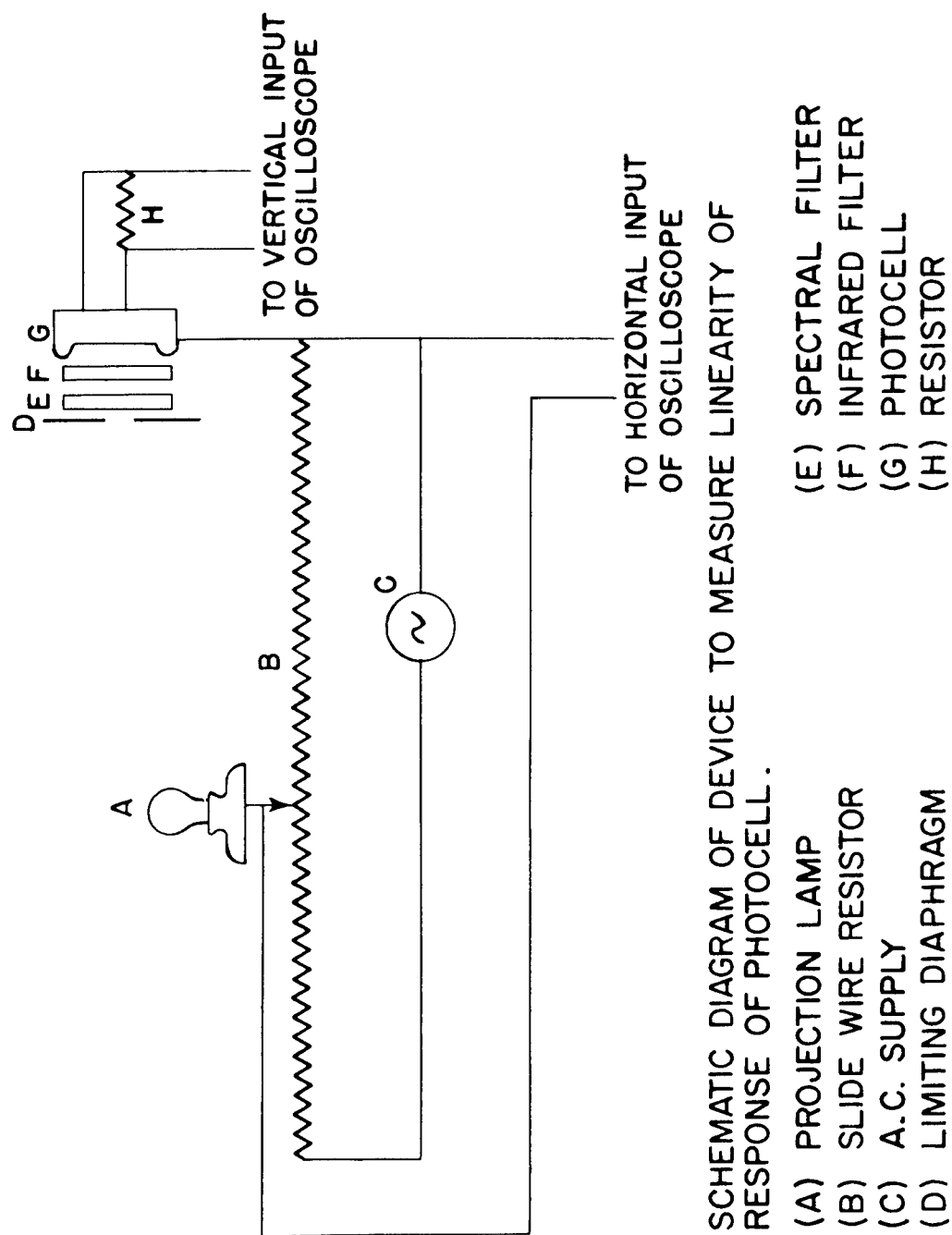


Figure 4. Linearity measurement apparatus.

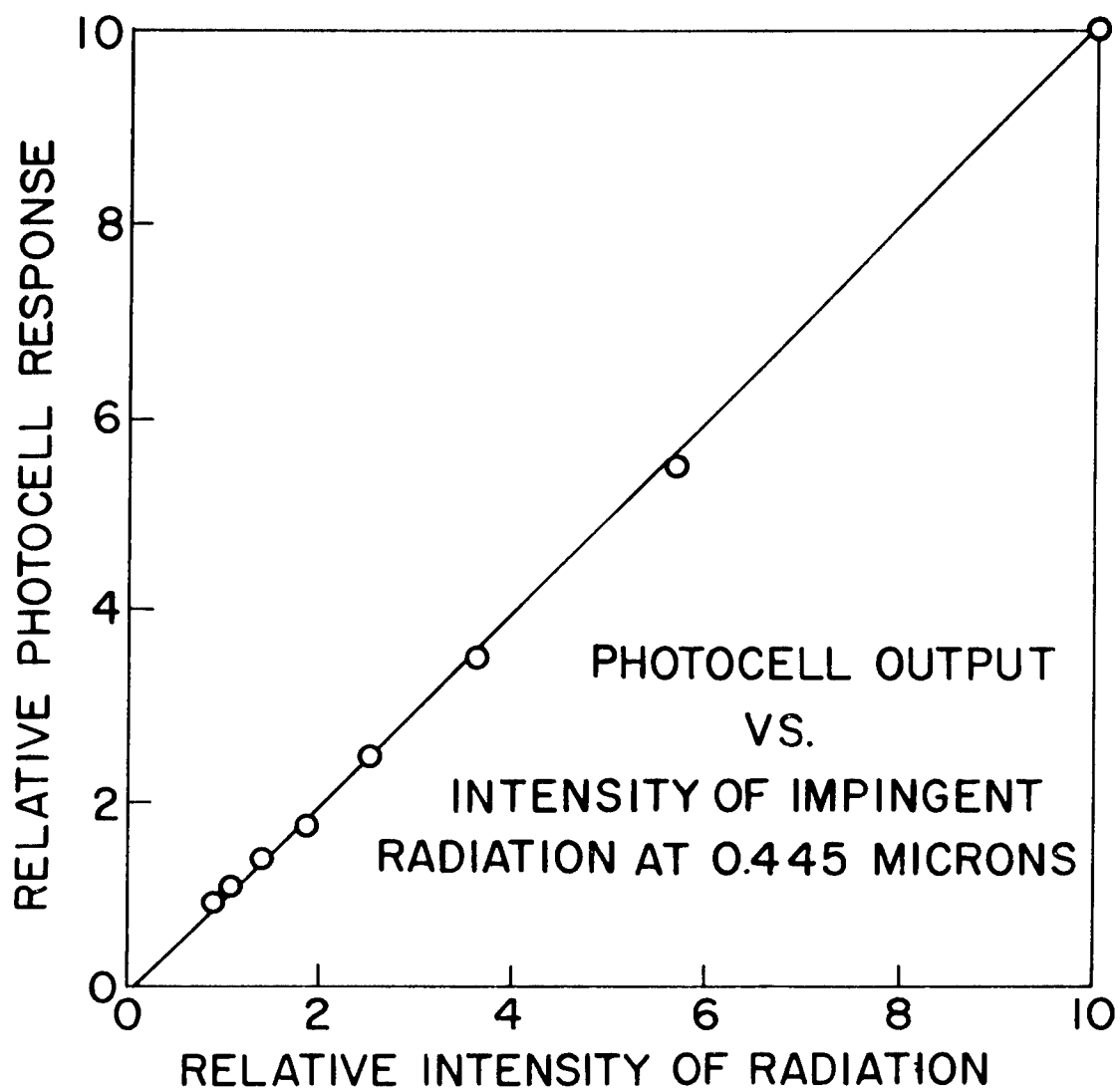


Figure 5. Linearity of photocell at 0.445 microns.

of the lamp filament. In each case the curve yielded an equation of the form:

$$V = a T^x$$

where

V = photocell output

T = brightness temperature $^{\circ}\text{K}$

a and x = constants dependent upon wavelength.

In each case these plots were made with the external circuit of the photocell containing the proper resistance to give a linear plot of intensity of impinging radiation versus output of the photocell. A typical calibration curve and the equation are given in Fig. 6. Figure 7 is a photograph of the equipment used in determining the temperature response of the photocell.

The spectral band pass of the various filters in combination with the infra-red filter was determined on a Cary Model 14 recording spectrophotometer. The results of this determination are given in Fig. 8.

PROCEDURE

Of the many available high purity ceramic oxides those of the lanthanum rare-earth series stand out in their refractory characteristics. The oxides of yttrium, samarium, gadolinium, erbium and lutetium are quite similar in physical properties and show great promise as super refractories. All of the oxides for which data are given in this report were prepared at the Ames Laboratory, Ames, Iowa.

Specimen Fabrication

The specimens (see Fig. 9) were formed around a thermocouple junction by dry pressing the powder oxides in a simple cylindrical die

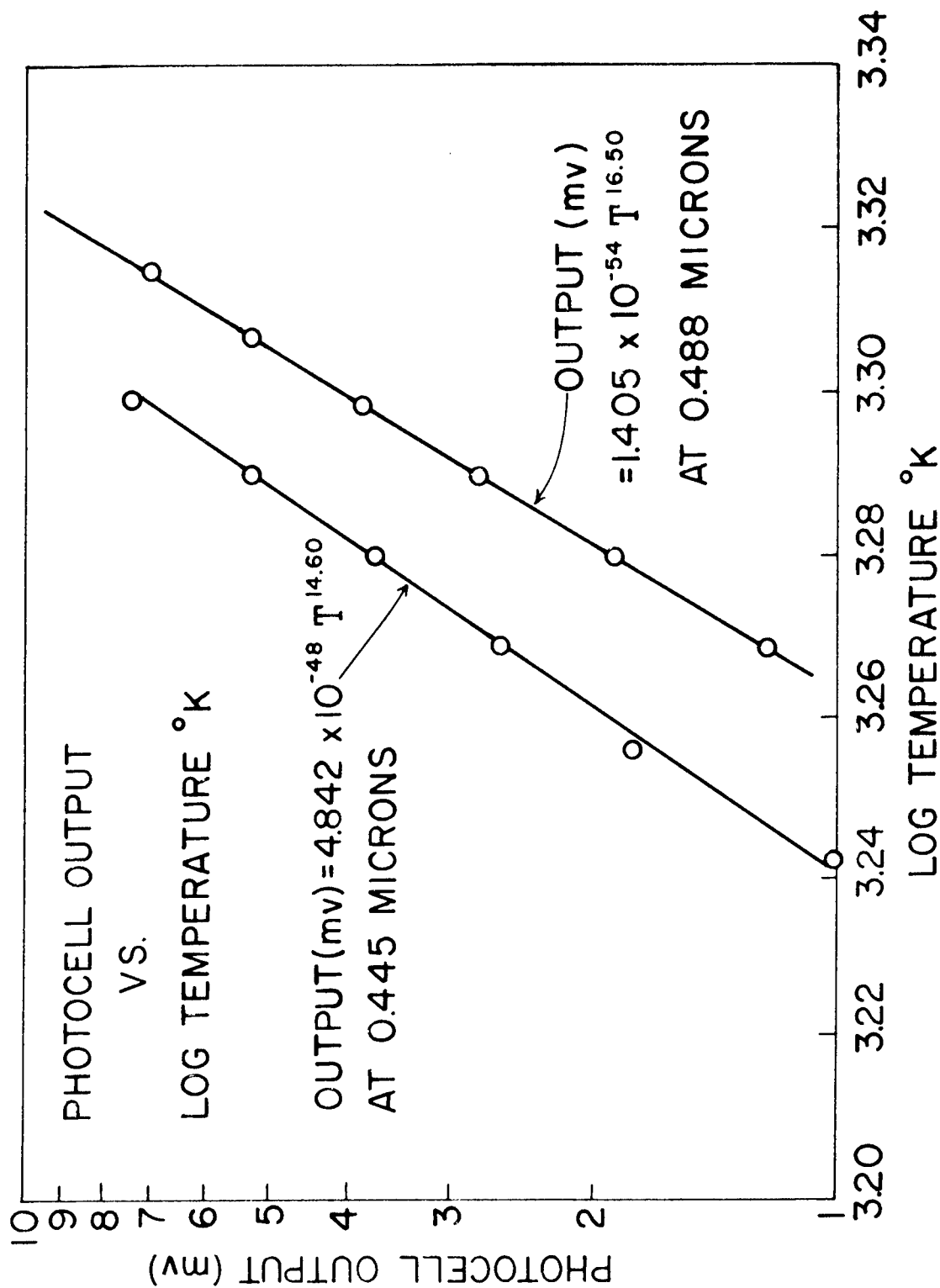


Figure 6. Brightness temperature response of photocell at 0.445 and 0.488 microns.

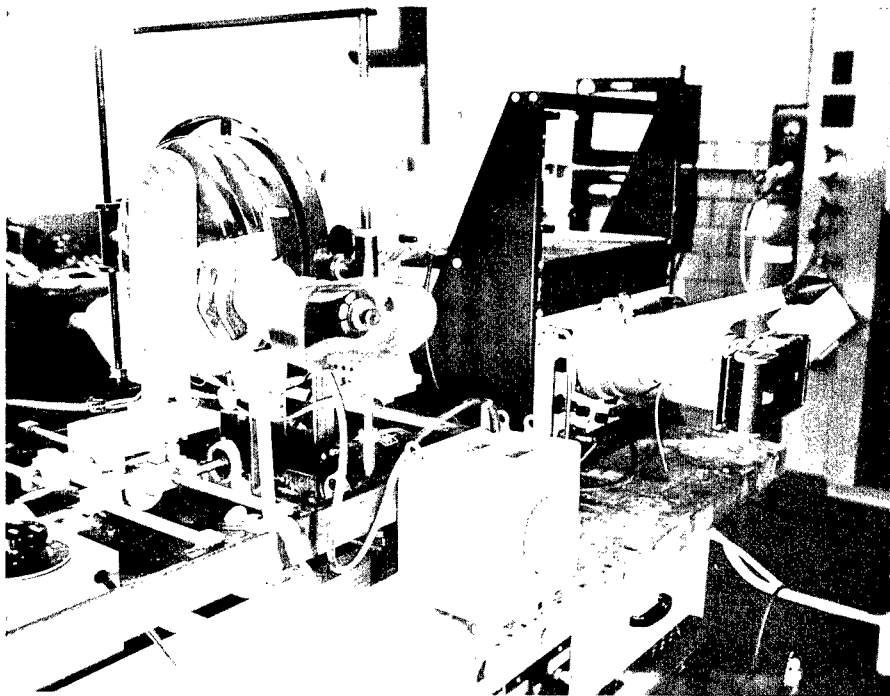


Fig. 7. Photograph of temperature response calibration equipment.

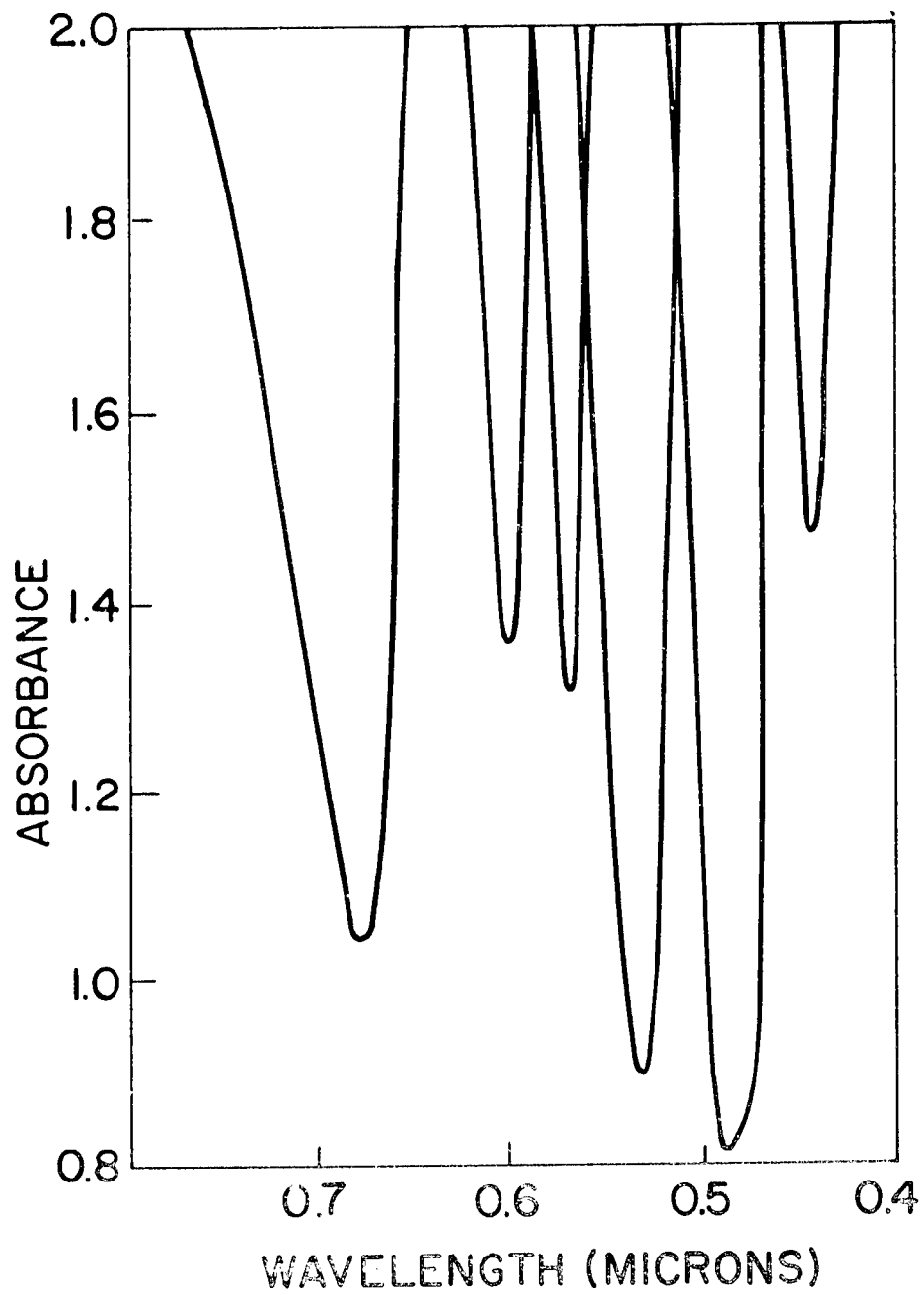


Figure 8. Absorbance versus wavelength for Wratten filters 70, 72B, 73, 74, 75 and 76.

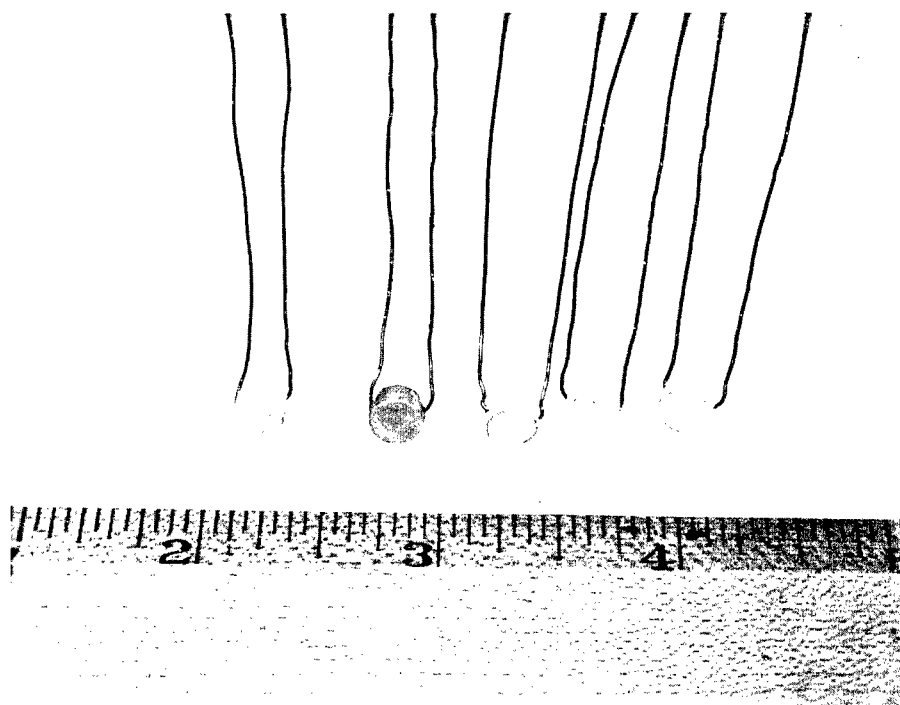


Fig. 9. Photograph of specimens.

with a fitted plunger. The plunger of the die was ground to give it two flat, parallel surfaces along its length. These flat surfaces allowed the thermocouple leads to extend out of the die when the plunger was inserted.

A fraction of the powdered material to be fabricated was placed in the die and lightly compressed by hand. The thermocouple was then bent and positioned so its junction would be at the center of the specimen and its leads would extend out of the die. Then the remainder of the powdered material was placed in the die. The plunger was inserted and the materials compressed under a pressure of approximately 75,000 lb. per sq. in.

The specimen was then removed from the die and fired in a platinum wound resistance furnace at 1500°C for a period of 24 hr. To avoid contamination of the specimen from the refractory tube of the furnace, it was supported by the thermocouple leads and completely isolated from all other refractory materials.

After cooling, the specimen was removed from the furnace and ground on each of its circular faces until it was approximately $1/8$ in. thick with the thermocouple at its center.

Measurement of Emissivity

A reflectance method was used to determine the emissivity of the oxides. All measurements of intensity of reflected radiation were relative rather than absolute.

The specimen was supported in the furnace at the focal point by the thermocouple leads with one of its faces perpendicular to the axis of the

furnace. The specimen holder was water cooled and could be rotated in a horizontal plane. The magnesium oxide smoked plate was located on one side of the specimen holder and was located at the specimen position when the holder was rotated 90° . The photocell could therefore "see" either the standard of reflectance or the specimen simply by rotating the specimen holder 90° .

The thermocouple leads were extended with compensating lead wire to a reference junction at 0°C , and then with copper leads to the oscilloscope. The photocell output was fed into the resistance box and the potential drop across the resistor was also then fed into the oscilloscope.

With spectral and infra-red filters in the optical path and the corresponding resistance in the external circuit of the photocell, an arc was struck between the carbons of the furnace. The magnesium oxide smoked plate was placed at the specimen position and a reading of output of the photocell, or reflectance, was taken. A second reading was then taken with the arc off. The output of the photocell in this case was the result of room lighting which was reflected by the standard reflector and was zero in all cases. These two measurements provided a calibration of the vertical trace of the oscilloscope; the upper point corresponding to a reflectance of 0.98 and the lower point 0.00.

The specimen was then rotated into the focal point and the arc struck. The oscilloscope trace was photographed with a time exposure yielding a plot of output of the photocell versus output of the thermocouple or, in physical values, intensity of combined reflected and emitted radiation versus temperature.

To determine the emissivity from reflectivity data it was necessary to find a method of separating the radiation emitted by the specimen from that reflected by it. If the brightness temperature response of the photocell is known and the true temperature and emissivity of the specimen are also known, the output of the photocell due to the radiation emitted by the specimen can be calculated by making use of Wien's equation:

$$\ln \epsilon_{\lambda} = c_2 / \lambda \left(\frac{1}{T} - \frac{1}{T_b} \right).$$

The calculation involves a method of successive approximations. The first approximation is to determine the approximate emissivity of the specimen from the reflectance curve by assuming that the radiation emitted by the specimen does not contribute to the output of the photocell. With this value, the approximate brightness temperature of the specimen can be calculated. If the brightness temperature response of the photocell is known, corrections can then be made in the emissivity value. By continuing this type of successive approximations the emissivity of the specimen can be determined with any desired degree of accuracy.

The entire procedure was repeated for each oxide and filter-resistor combination with a new standard of reflectance prepared before each determination.

RESULTS

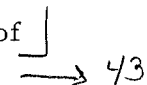
It was noted that the output of the photocell, when viewing the specimens during an emissivity determination, was never less than 20 millivolts. The minimum output occurred when erbium oxide at approximately

800°C was viewed through the 0.680 micron filter. When this was compared with the output of the photocell when viewing the tungsten filament lamp at 800°C through the same filter (less than one-half millivolt), it could be seen that the contribution of the radiation emitted by the specimen toward the total output of the photocell was negligible. Further investigation showed that in no instance did the emitted radiation account for this large portion of the total output. For this reason the method of successive approximations need not be continued beyond the first approximation to determine emissivity from the curves of combined reflected and emitted radiation versus temperature. The photographs of the oscilloscope traces may therefore be considered as plots of reflectivity versus temperature.

Data from the oscilloscope traces were recorded and changed to emissivity data simply by subtracting the reflectivity from unity. These data were then replotted on linear temperature scales. Curves of emissivity versus temperature for the various oxide-filter combinations are given in Figs. 10 through 14. Curves of emissivity versus wavelength at selected temperatures for the oxides are given in Figs. 15 through 19.

DISCUSSION OF RESULTS

A trend exists in the emissivities of ceramic oxides which is not followed by these results. In MgO, BeO, and Al_2O_3 an increase in spectral emissivity with increasing temperature is noted but abrupt changes such as those found herein are not commonly seen. It is interesting, therefore, to speculate upon the reasons for the existence of these anomalies.

The emissivity versus temperature curves for all of the oxides investigated except Sm_2O_3 exhibit a definite break in the direction of  43

CeO

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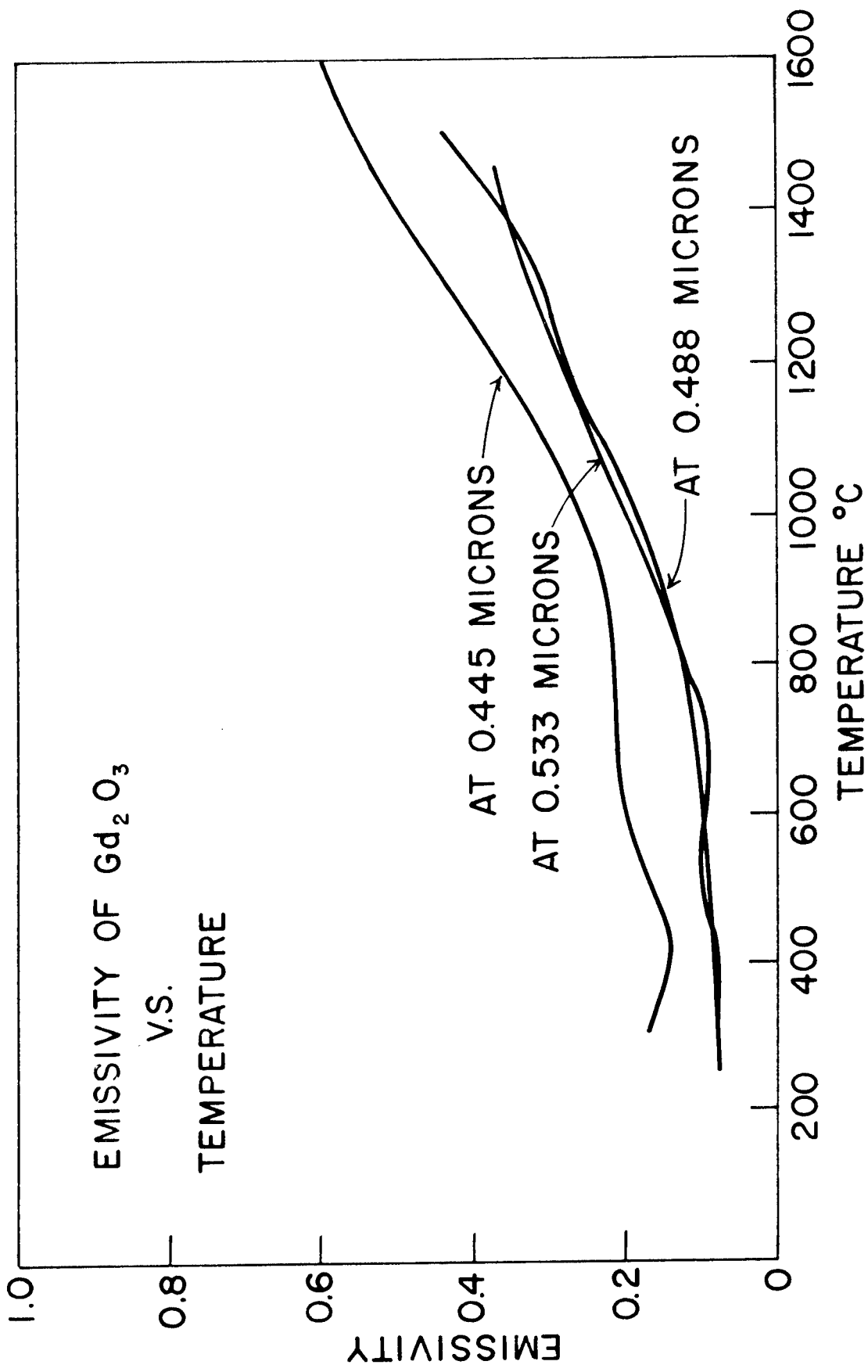


Figure 10. Emissivity versus temperature curves for Gd_2O_3 .

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CeO

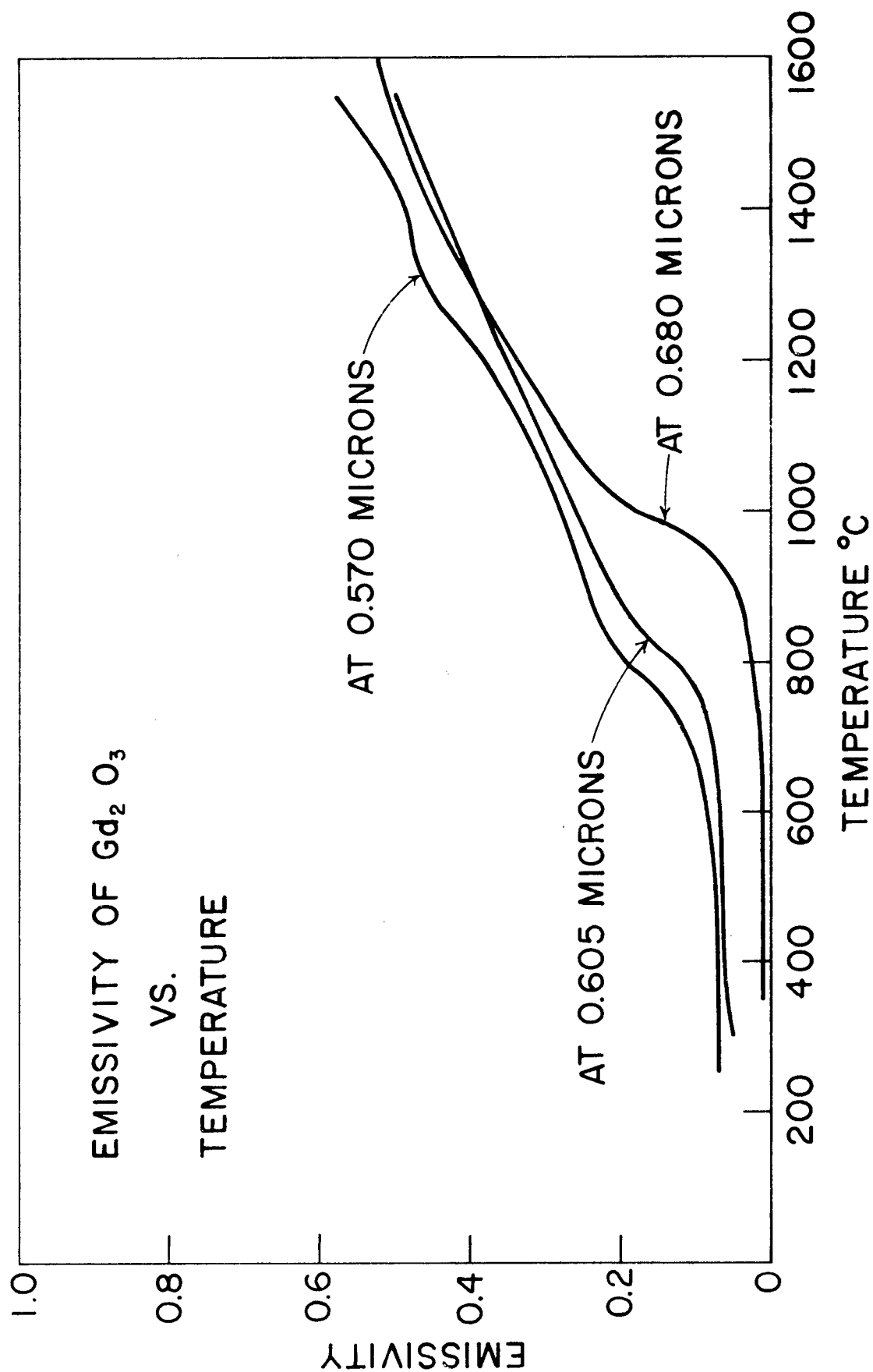


Figure 10. (Continued)

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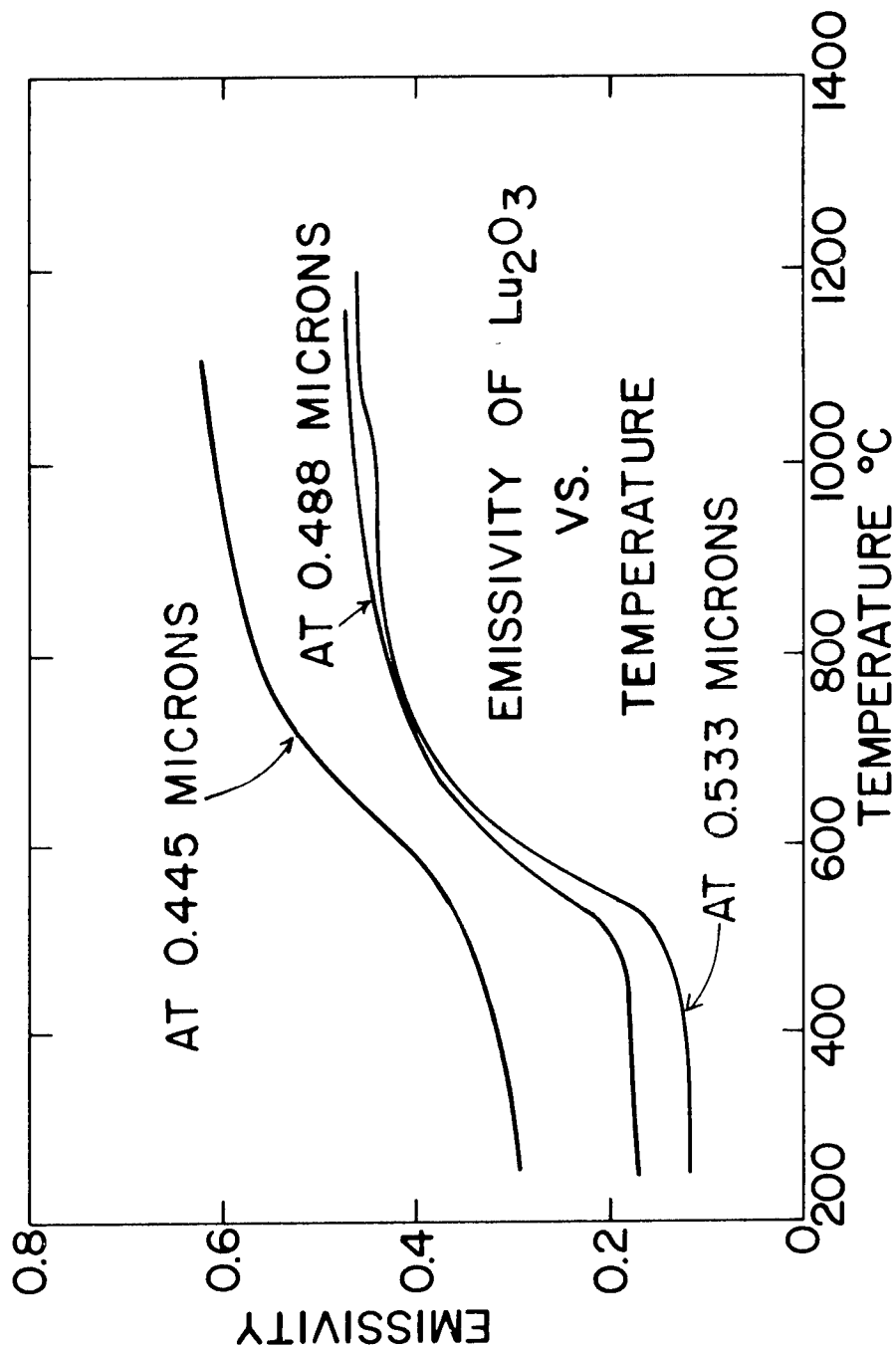
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Figure 11. Emissivity versus temperature curves for Lu_2O_3 .

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CeO

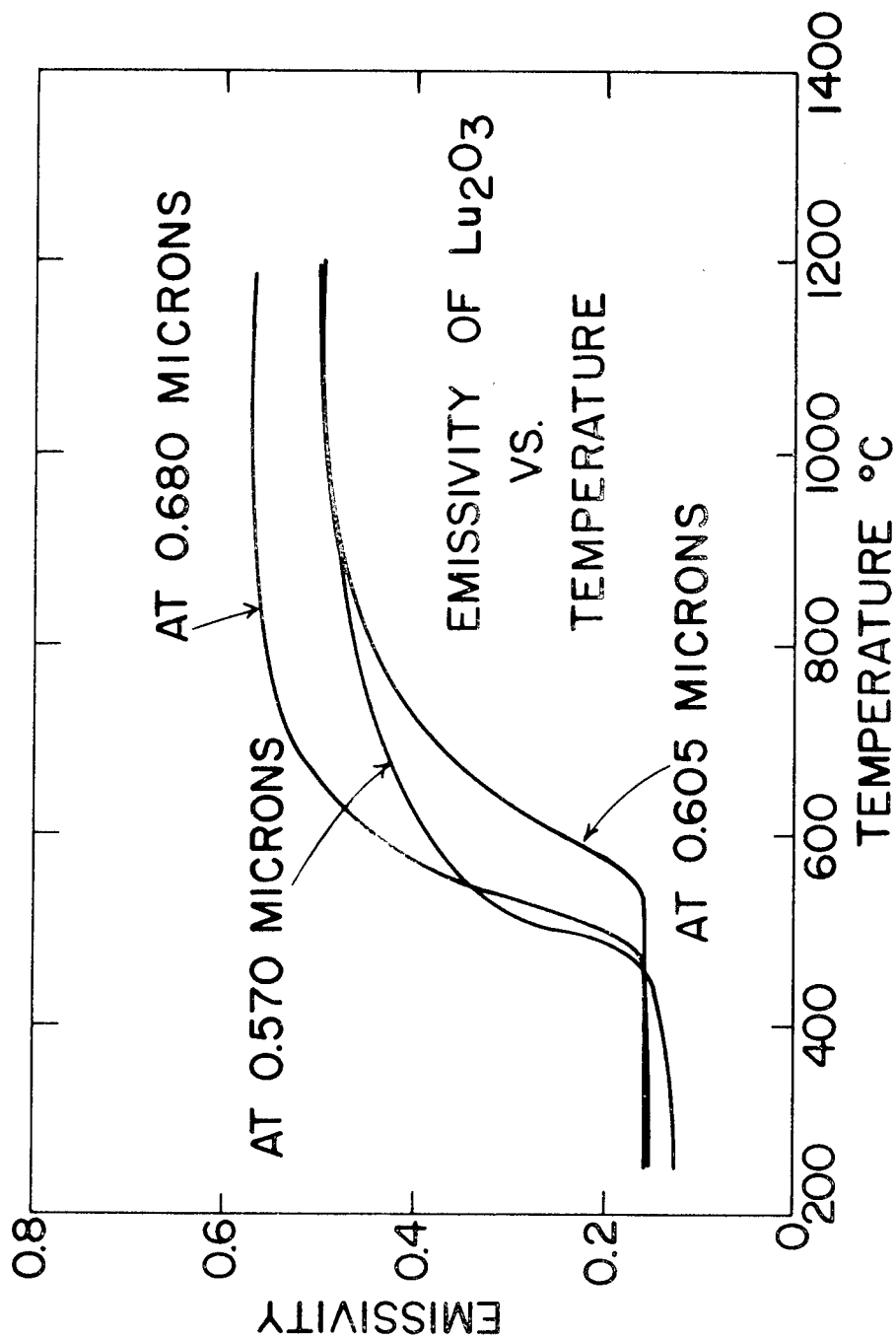


Figure 11. (Continued)

Red

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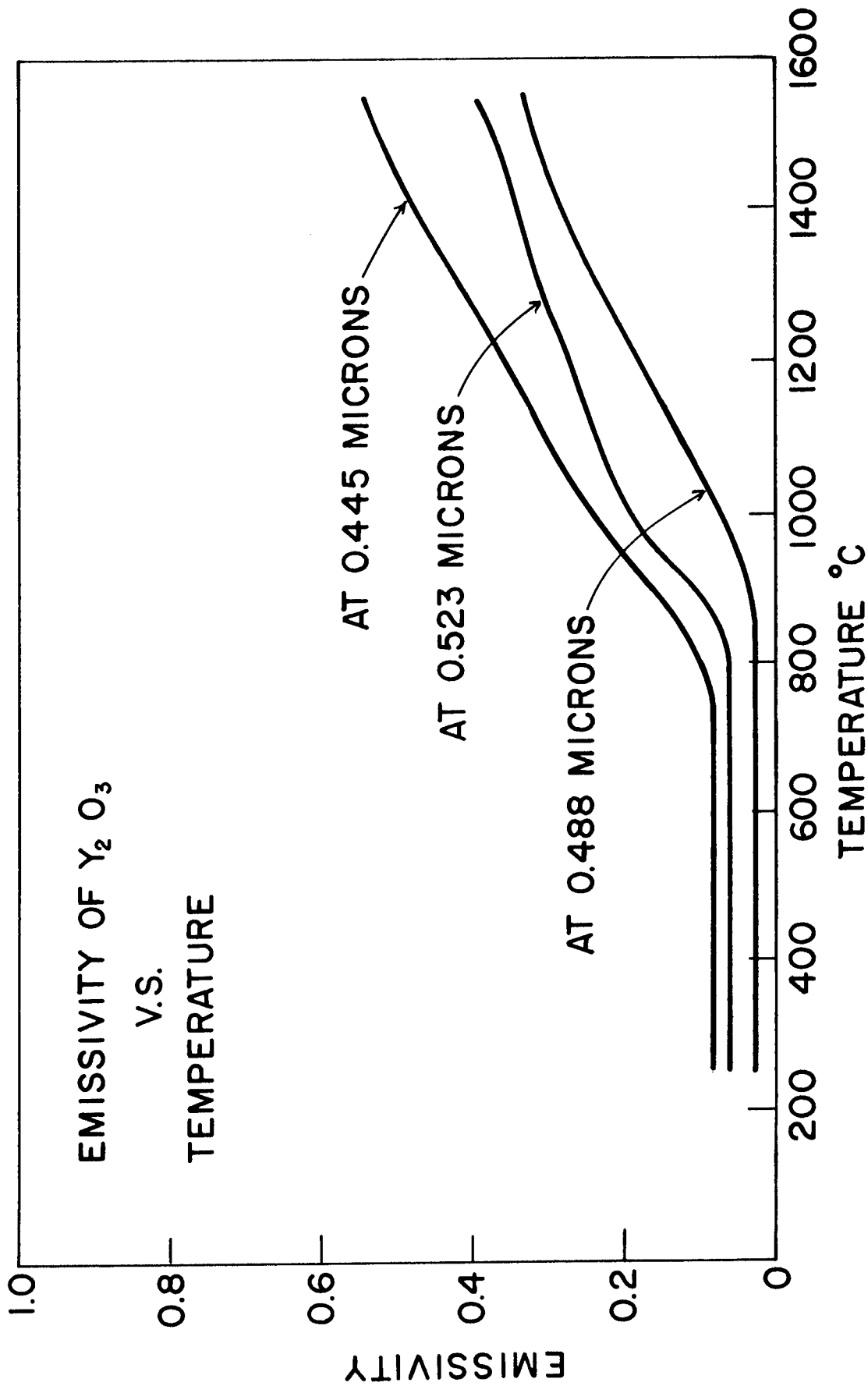


Figure 12. Emissionity versus temperature curves for Y_2O_3 .

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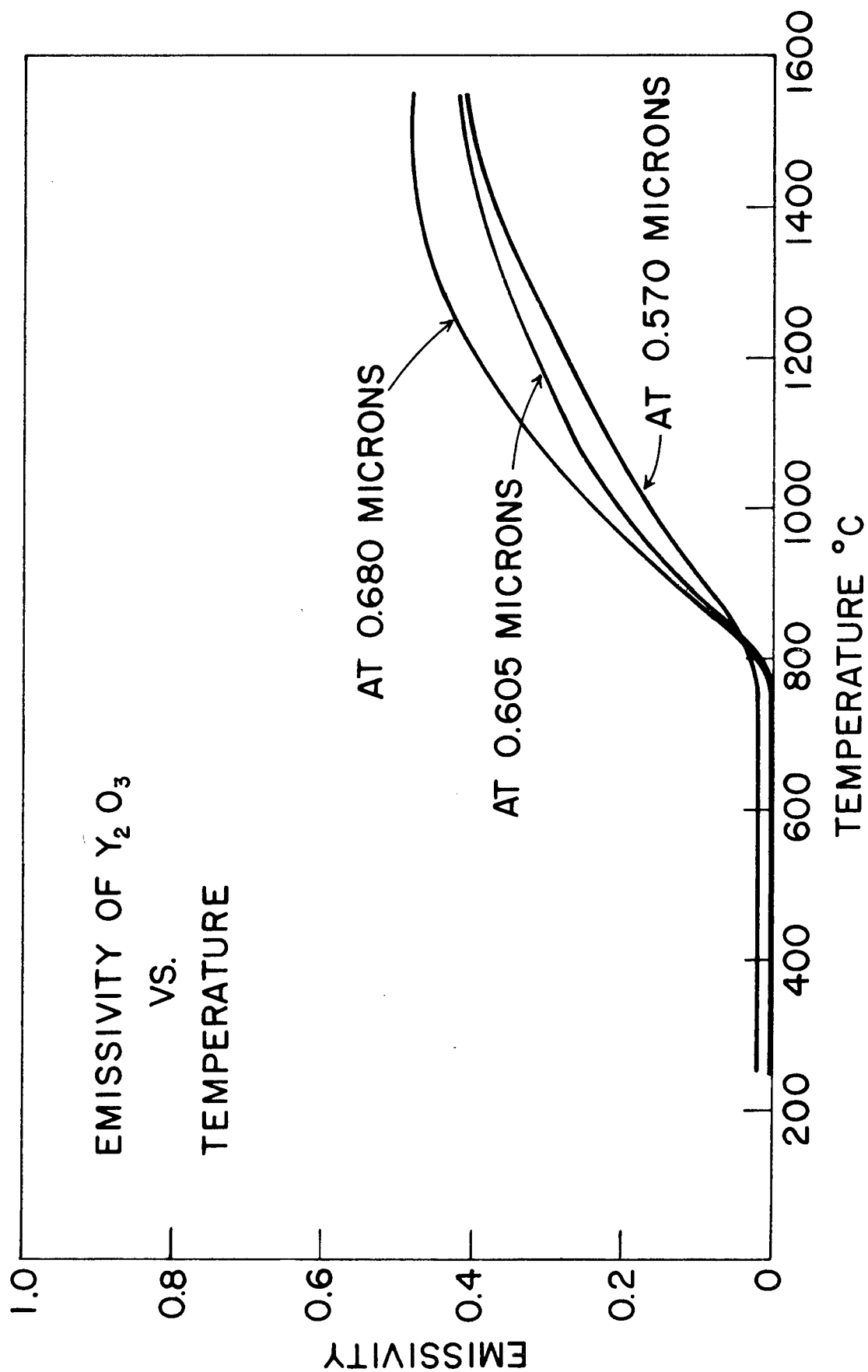


Figure 12. (Continued)

CeO

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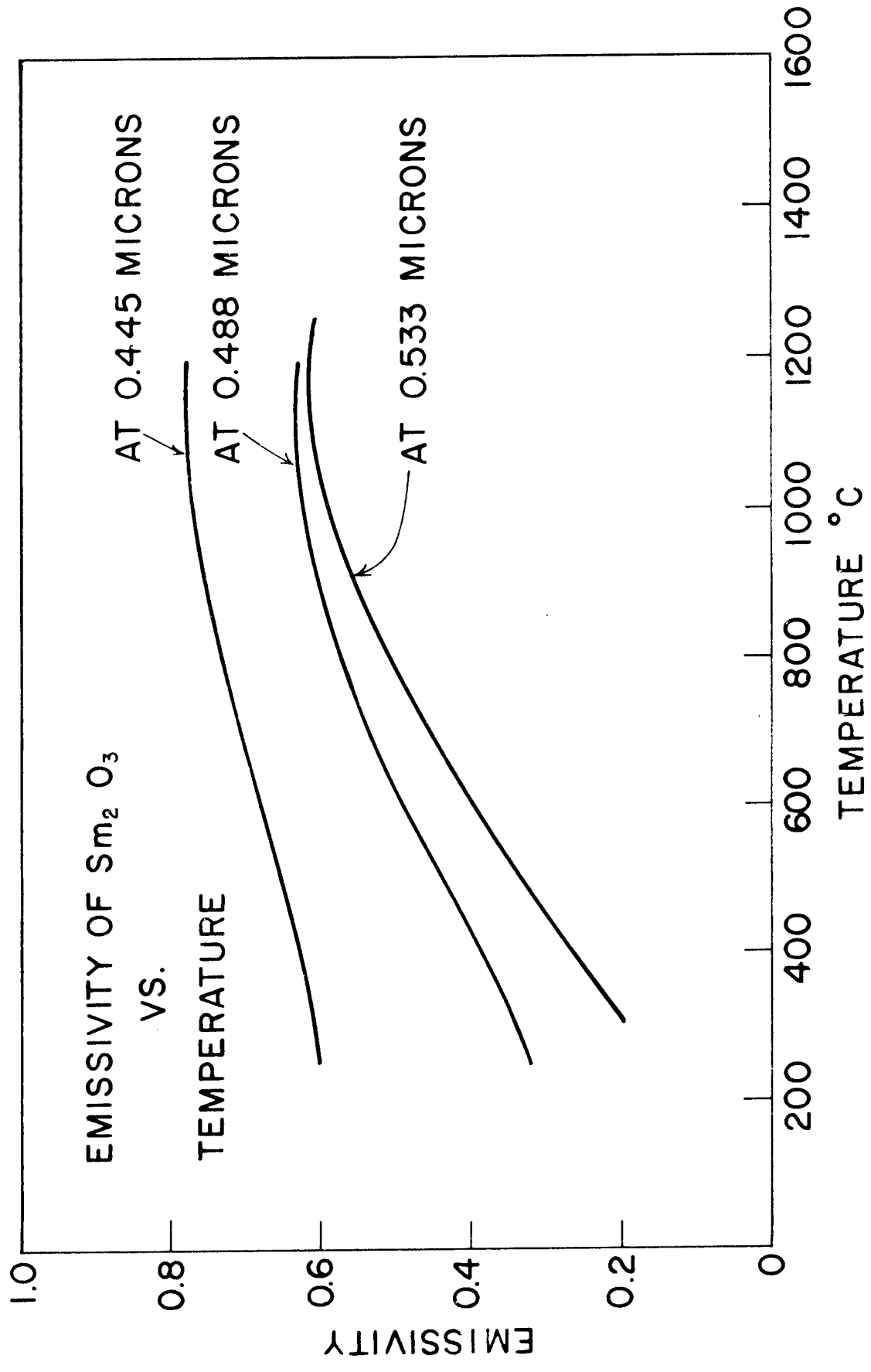


Figure 13. Emissivity versus temperature curves for Sm_2O_3 .

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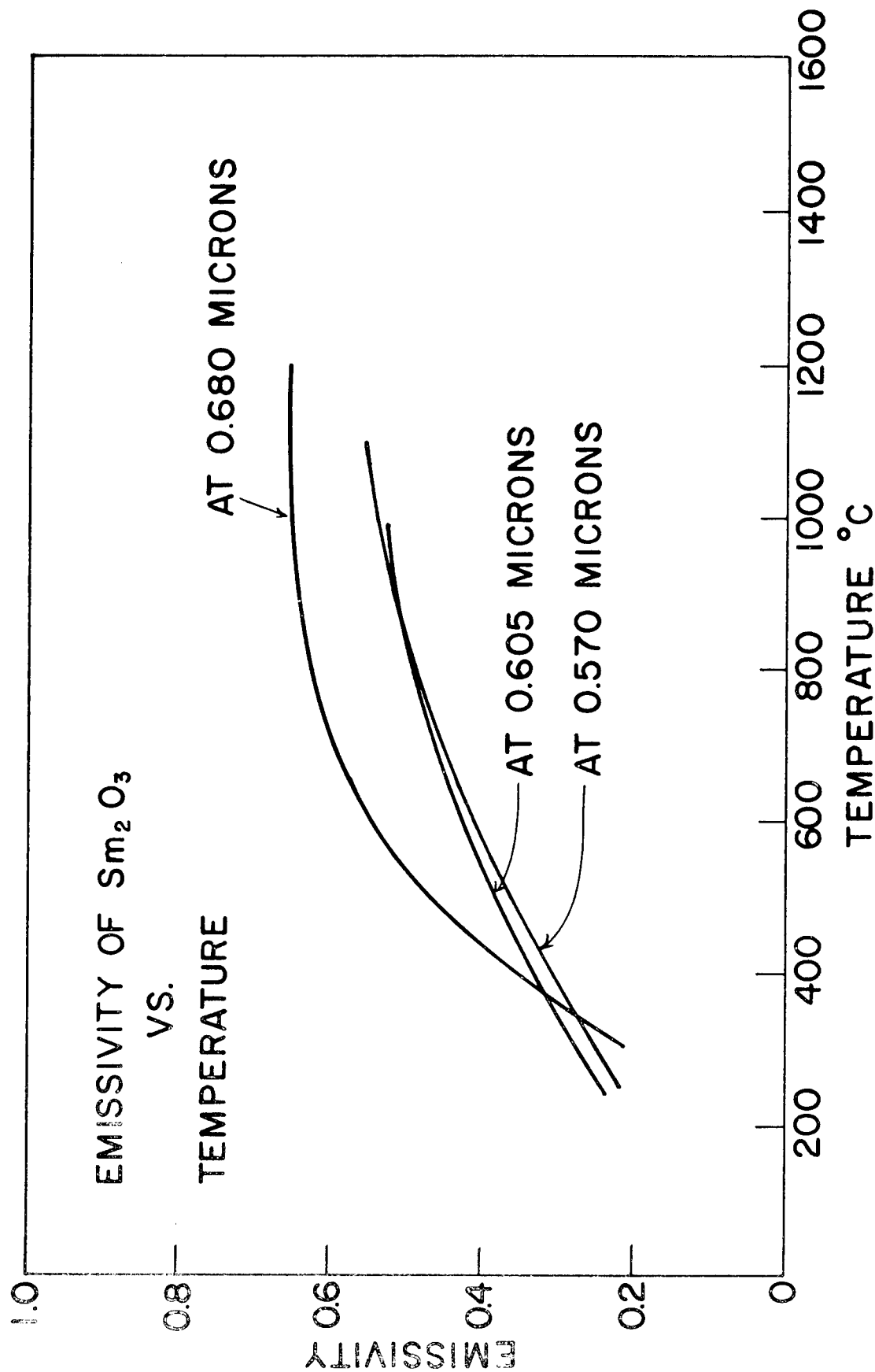


Figure 13. (Continued)

CeO

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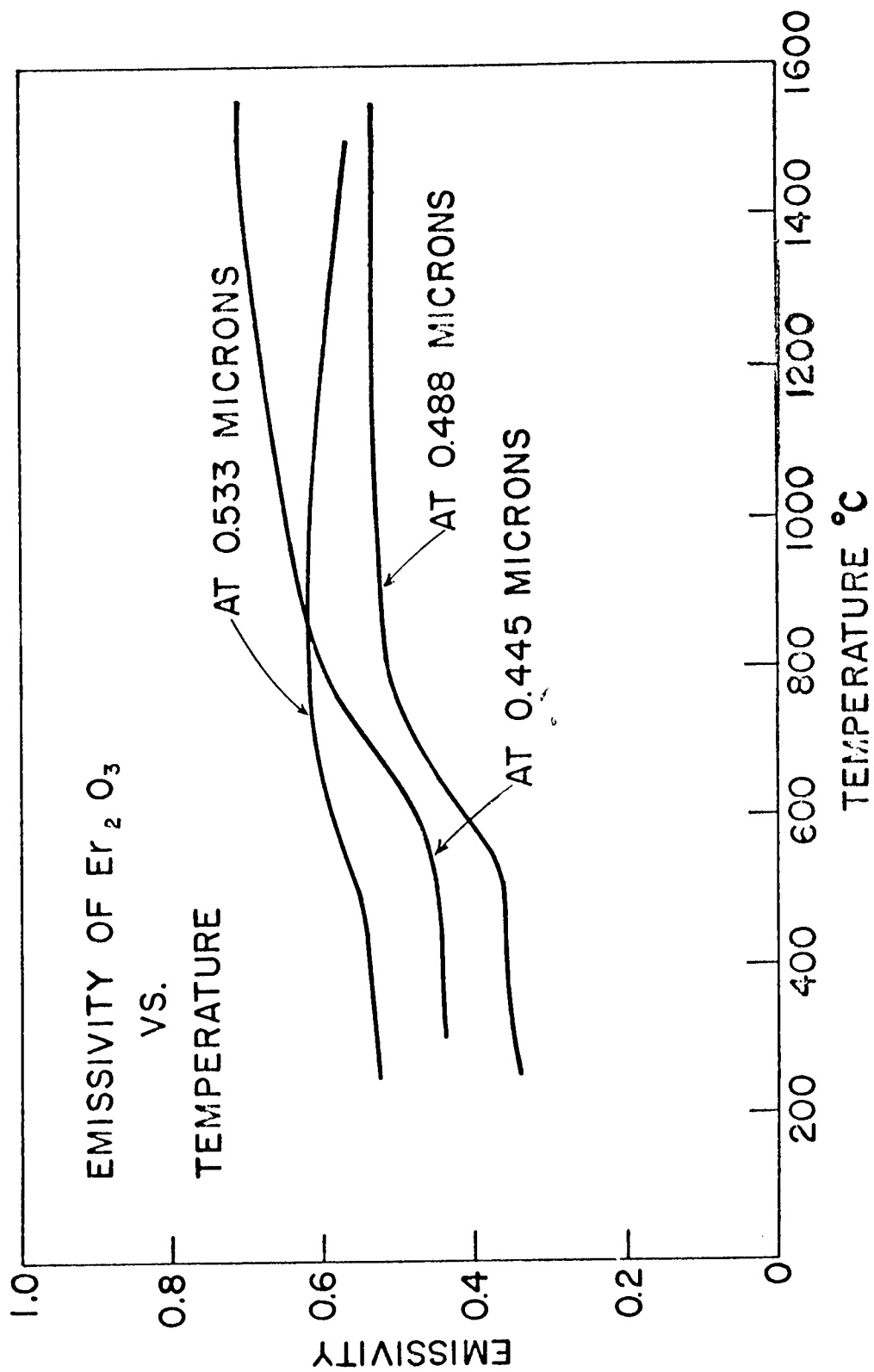


Figure 14. Emissivity versus temperature curves for Er_2O_3 .

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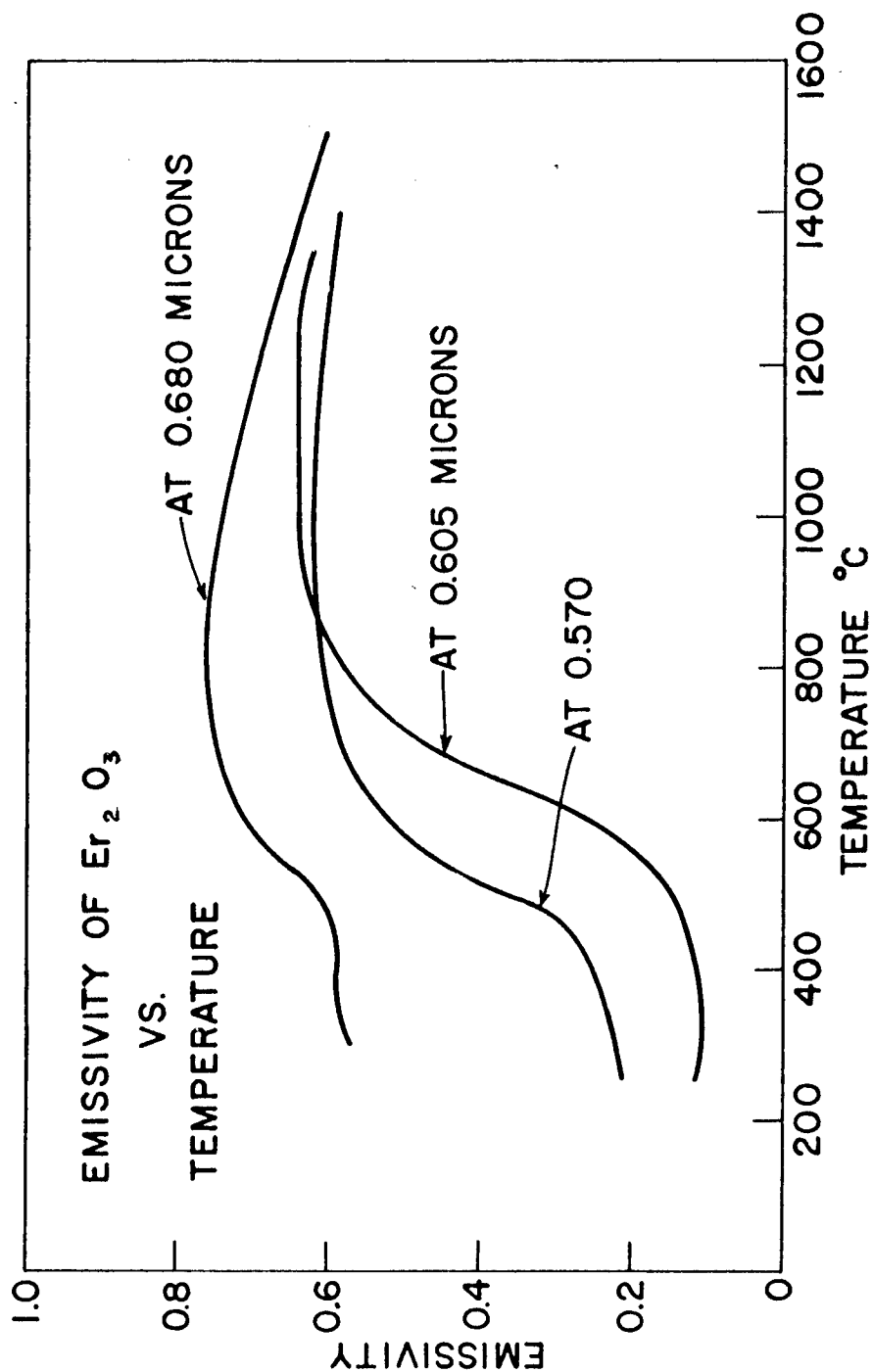


Figure 14. (Continued)

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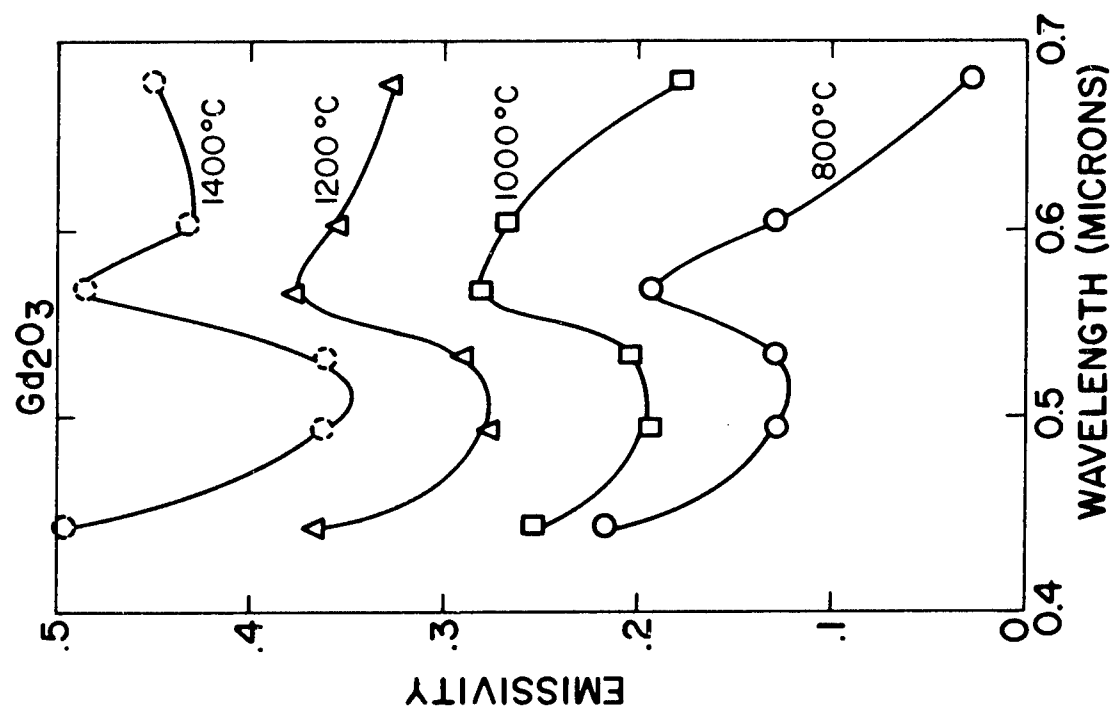


Figure 15. Emissivity versus wavelength curves for Gd_2O_3 .

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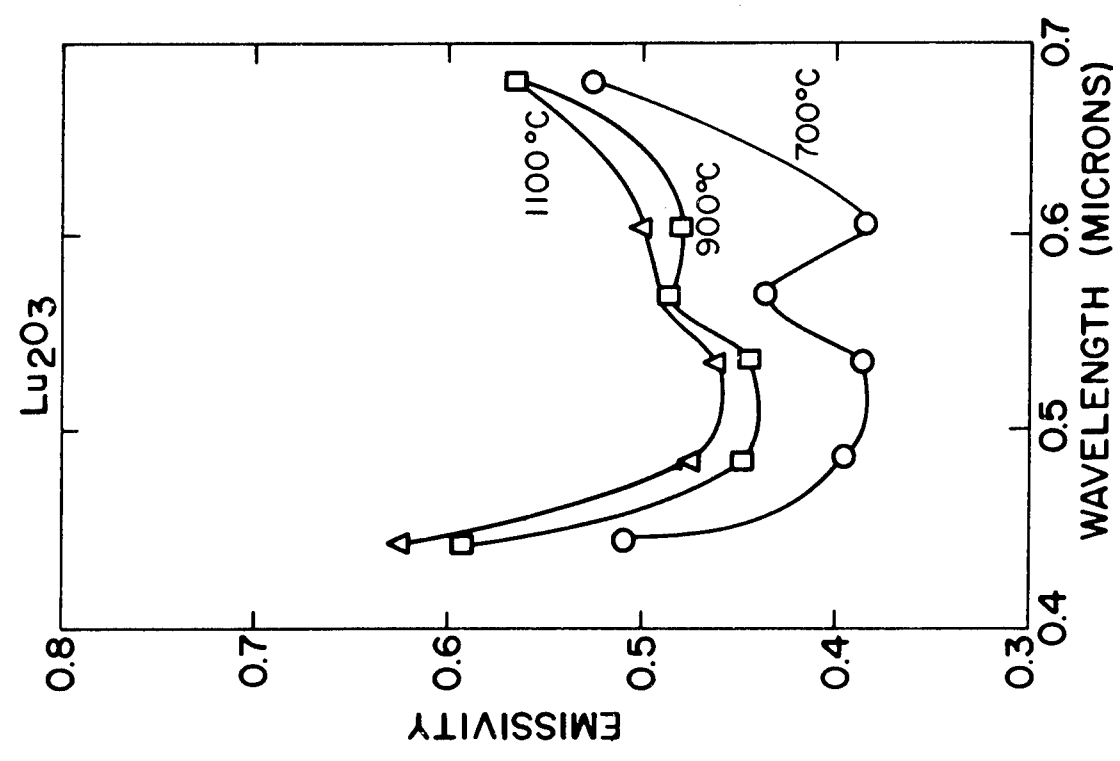


Figure 16. Emissivity versus wavelength curves for Lu_2O_3 .

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52548

Q₂₀

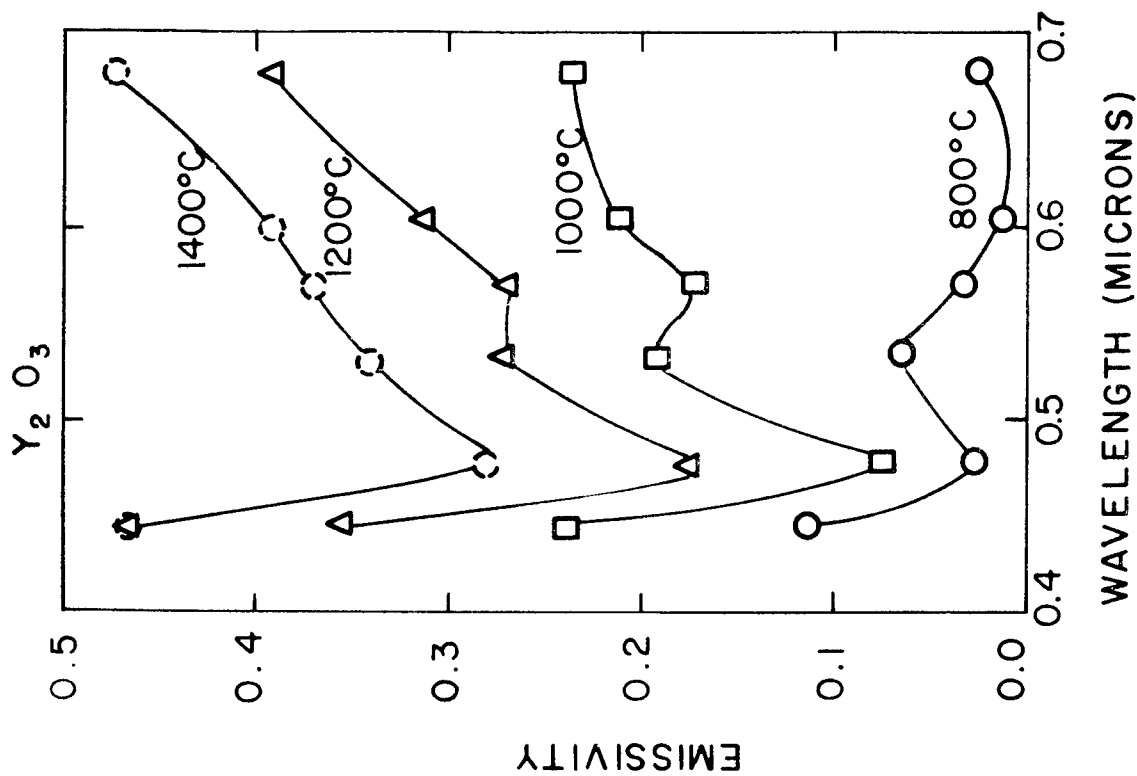


Figure 17. Emissivity versus wavelength curves for Y_2O_3 .

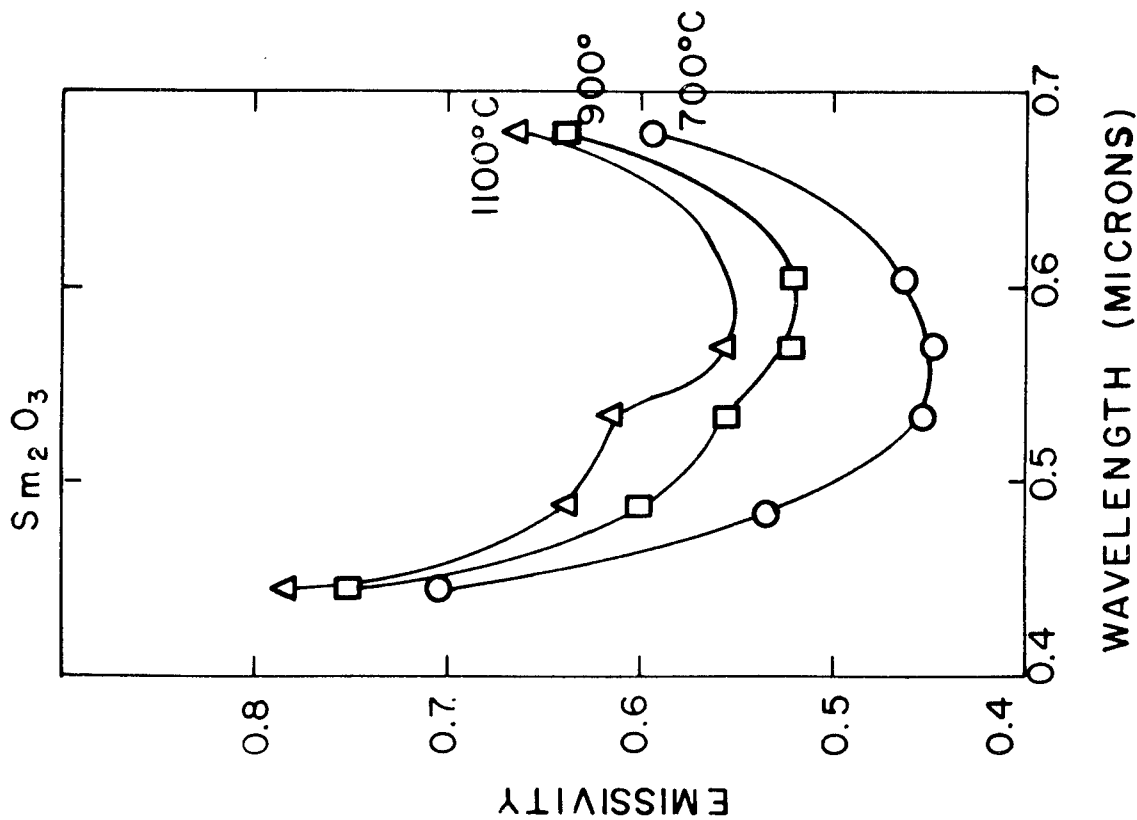


Figure 18. Emissivity versus wavelength curves for Sm_2O_3 .

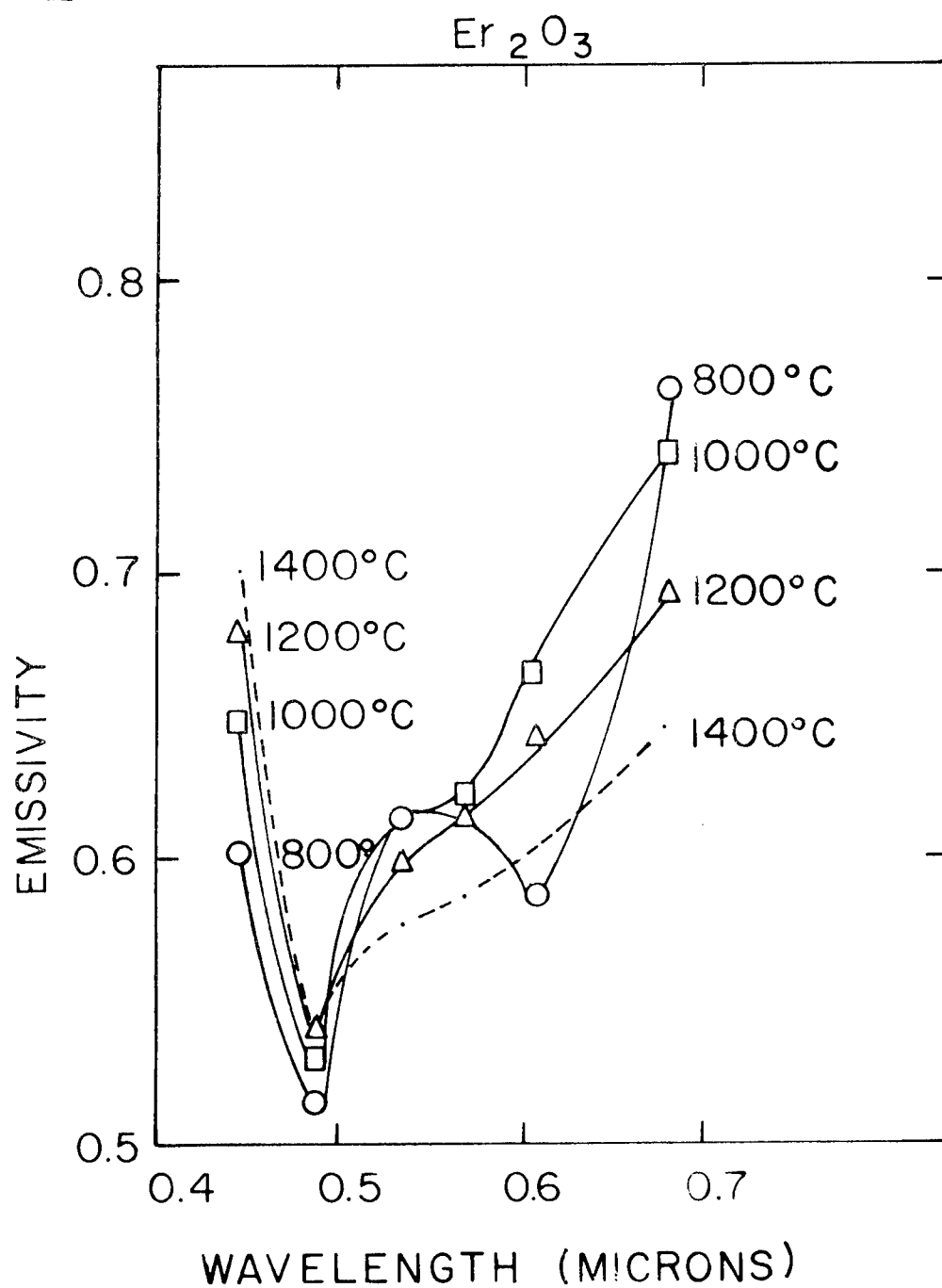


Figure 19. Emissivity versus wavelength curves for Er_2O_3 .

increasing emissivity at some temperature below 1000°C . This seems to indicate thermal initiation of some internal mechanism causing greater absorptance. Y_2O_3 is known to become an electronic conductor at a temperature somewhat above 700°C and this transition (from an insulator to an electronic conductor) in electrical properties might account for the shapes of the curves. When sufficient thermal energy is added to the atomic structure to excite some of the electrons to a conduction band, the electrons are free to be accelerated by the impinging radiation. It may be that at the temperatures indicated by the breaks in the emissivity curves, the impinging radiation is absorbed and its energy dissipated in accelerating the free electrons created by a transition in the electrical properties of the oxides.

The emissivity versus wavelength curves for Lu_2O_3 and Y_2O_3 are also of interest. At wavelengths of 0.570 and 0.533 microns, respectively, the curves seem to indicate the existence of an activation peak. At low temperatures inflections are noted in the emissivity curves at these wavelengths and as the temperatures of the oxides increase the inflections become less pronounced and finally disappear. This also indicates that there may exist a thermally initiated mechanism causing a change in the emissivity of the materials.

The sources of errors in this determination are few in number and small in effect. Surface condition of the specimen is a source of error in all determinations of emissivity since it is impossible to maintain an optically smooth surface at elevated temperatures because of thermal etching. The rapidity of the method used minimized this effect. The errors in all emissivity measurements due to nonopacity are also

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minimized by this method. Heating, illumination and viewing of the specimen are all performed on one surface, thus no radiation reaching the photocell is transmitted by the specimen. Any radiation which might be transmitted by the specimen would be absorbed if the specimen were thicker and would in no way change the reflectance measurements. The largest source of error is the discrepancy between the true temperature of the surface of the specimen and the temperature indicated by the thermocouple. This thermal gradient is minimized by using very small specimens.

There are two obvious extensions to this highly successful method. Replacement of the spectral filters with a monochromator would provide continuous emissivity versus wavelength data. Interchangeable photoelectric detectors, each with a different spectral sensitivity, would give a broader range of spectral data. These investigations are in progress.

SUMMARY

A method of determining the normal spectral emissivity of ceramic oxides has been developed. A carbon arc image furnace is used as a heat source and intensities of radiation are measured with a blocking-layer photoelectric cell. A method of successive approximations is used to determine emissivity from a curve of combined reflected and emitted radiation versus temperature.

The spectral emissivities of yttrium, samarium, gadolinium, erbium and lutetium oxides at six wavelengths in the visible spectrum have been determined at temperatures up to 1600°C.

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